



Government Of India Patent Office Todi Estates, 3<sup>rd</sup> Floor, Lower Parel (West) Mumbai – 400 013

## THE PATENTS ACT, 1970

IT IS HEREBY CERTIFIED THAT, the annex is a true copy of Application and Provisional Specification filed on 21/11/2003 in respect of Patent Application No.1202/MUM/2003 of Reliance Industries Limited, an Indian Company, having its Registered office at Maker Chambers IV, 3<sup>rd</sup> Floor, 222, Nariman Point, Mumbai 400 021, Maharashtra, India.

This certificate is issued under the powers vested in me under Section 147 (1)

of the Patents Act, 1970.

Dated this | 8 Inday of Jaman 2005.

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#### FORM I

### THE PATENTS ACT, 1970 (39 of 1970)

#### APPLICATION FOR GRANT OF A PATENT

(See sections 5 (2), 7, 54 and 135; rule 39)

- We, Reliance Industries Limited, an Indian Company, having its Registered Office at Maker Chambers IV, 3<sup>rd</sup> Floor, 222, Nariman Point, Mumbai 400 021, Maharashtra, India
- 2. Hereby declare:
  - a) that we are in possession of an invention titled " Modified Polyethylene Terephthalate For Low Temperature Dyeability And Controlled Shrinkage"
  - b) that the provisional specification relating to this invention is filed with this application.
  - c) that there is no lawful ground of objection to the grant of patent to us;
- 3. further declare that the inventor(s) for the said invention are:
  - a) Dr. Vikas. M. Nadkarni, residing at A -18, Garden Estate, Off D. P. Road, Aundh, Pune 411007, Maharashtra, India
  - b) Anjan K Mukhopadhyay , residing at A-48, Reliance Housing Complex, Mohapada, Rasayani, Dist. Raigad Pin. 410222
    Maharashtra, India
  - c) Ashwin. K Jain, residing at C-6/2/1/1, Sector –18
    Cidco Colony, New Panvel, Navi Mumbai 410206, Maharashtra, India

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d) Manoj Jhaver residing at C/O Mr. Ashok Chandak, B –607, Dheeraj Pooja, Chincholi Bunder Road, Malad (West), Mumbai Mumbai, 400 064, Maharashtra, India

all citizens of India.

That we are the assignees of the true and first inventors.

That our address for service in India is:
Reliance Industries Ltd., Polyester Sector,
RPL House, 15 Ballard Estate, Mumbai – 400 038
Attn.: Mr. Anup K. Rakshit

Following declaration was given by the inventors

We,

- a) Dr. Vikas. M. Nadkarni, residing at A -18, Garden Estate, Off D. P. Road, Aundh, Pune 411007, Maharashtra, India
- b) Anjan K Mukhopadhyay , residing at A-48, Reliance Housing Complex, Mohapada, Rasayani, Dist. Raigad Pin. 410222
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  Cidco Colony, New Panvel, Navi Mumbai 410206, Maharashtra, India
- d) Manoj Jhaver residing at C/O Mr. Ashok Chandak, B –607, Dheeraj Pooja, Chincholi Bunder Road, Malad (West), Mumbai Mumbai, 400 064, Maharashtra, India

all citizens of India.

The true and first inve	ntors for this inventi-	on declare that	the applicants	herein
are our aşsignee.			• •	

Dr. Vikas. M. Nadkarni

Alem the padhyay Anjan K Mukhopadhyay

Ashwin, K Jain

Manoj Jhaver

That to the best of our knowledge, information and belief the fact and matters stated herein are correct and that there is no lawful ground of objection to the grant of patent to us on this application.

Following are the attachment with the application:

- a. Provisional Specification (3 copies)
- b. Form 2.
- c. Form 3.
- d. Abstract.
- e. Fee Rs 3000 by cheque

We request that a patent be granted to us for the said invention.

Dated this 21st day of November, 2003.

FOR RELIANCE INDUSTRIES LTD

President

(Sh.S.P. SAPRA)

To:

The Controller of Patents
The Patent Office

Mumbai.

#### FORM 2

## THE PATENTS ACT, 1970 (39 of 1970)

## PROVISIONAL SPECIFICATION SECTION 10

"MODIFIED POLYETHYLENE TEREPHTHALATE FOR LOW TEMPERATURE DYEABILITY, CONTROLLED SHRINKAGE CHARACTERISTICS AND IMPROVED TENSILE PROPERTIES"

RELIANCE INDUSTRIES LTD an Indian Company having its Registered office at Maker Chambers IV, 3<sup>rd</sup> Floor, 222 Nariman Point, Mumbai 400 021, Maharashtra, India.

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is performed:-

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MODIFIED POLYETHYLENE TEREPHTHALATE FOR LOW TEMPERATURE DYEABILITY, CONTROLLED SHRINKAGE CHARACTERISTICS AND IMPROVED TENSILE PROPERTIES

### Field of the Invention

The invention relates to modified polyethylene terephthalate polymers and fibers, yarns and fabrics produced therefrom having low temperature dyeability and controlled shrinkage characteristics.

## Background of the Invention

Polyester fibers are strong and lightweight and generally have good elastic memory. Polyester fabrics resist wrinkles and retain their shape in use. Polyester fibers are often blended with cotton as well as other fibers to produce fabrics, particularly for clothing.

Unmodified polyethylene terephthalate (PET) can generally only be dyed with disperse dyes at elevated temperatures of about 130° C and under high-pressure. Such high temperatures and pressures require high temperature high-pressure (HTHP) dyeing equipment. The need for such specialized dyeing equipment requirement is a principal reason for the limited usage of such PETs. This is particularly so in the decentralized sectors of hand looms and power looms, where such HTHP dyeing equipment is generally not available.

It is also known in the art to improve the dyeability of polyester fibers by using a dye carrier. The use of a dye carrier, however, leads to poor color fastness, and the dye carrier is retained in the dye processing wastewater. This presence of the dye carrier in the wastewater effluent is a pollutant and is anti-environmental. The art desired a readily dyeable PET, particularly suitable in the handloom and power loom sectors of the industry, and which avoided the use of dye carrier.

It is known to modify polyesters by polyethylene glycol (PEG). The PEG modified PET provides some improvement in dye uptake. Generally, polyethylene glycol modified polyethylene terephthalate provides copolymer polyester fibers which cause difficulties in weaving and fabric finishing due to inferior tensile strength. The art desires a PET fiber with improved dyeability, particularly at relatively low temperatures and atmospheric pressures, as well as controlled shrinkage and tensile properties.

The present invention provides the solution to the aforesaid prior art needs.

## Summary of the Invention

It is a principal object of the present invention to provide a modified polyethylene terephthalate fiber that possesses low temperature dyeability.

It is a further object of the present invention to provide a modified polyethylene terephthalate fiber and yarn with controlled shrinkage.

It is still a further object of the present invention to provide modified polyethylene terephthalate fibers as aforesaid with improved processing characteristics and particularly suitable for handloom and power loom operations.

It is still a further object of the present invention to provide a method for producing the aforesaid modified polyethylene terephthalate.

It is yet a further object of the present invention to provide the method for producing the modified PET as aforesaid, which method avoids the use of prior art dye carrier.

In one aspect, the present invention is a modified PET having low temperature dyeability and controlled shrinkage.

In another aspect, the present invention is a novel copolyester produced by polymerization of a terephthalic acid or its ester, an ethylene glycol, a flexible long chain aliphatic dicarboxylic acid or its ester, and a hydroxy terminated polyether polyol (mol. wt. range of 200 to 2000)

In still another aspect, the present invention is a modified PET disperse dyed yarn or fabric having a dye index greater than 100 when homopolymer PET is dyed at 100°C, and preferably at least about 120-500.

The draw twisted yarn of modified PET of the present invention exhibits a controlled shrinkage of 6% to 10 %.

The present modified PET copolymer of the present invention may be formed by batch or continuous polymerization.

## Description of the Invention

The copolymer of the present invention comprises a terephthalic acid or its ester equivalent in combination with an ethylene glycol, a flexible long chain aliphatic dicarboxylic acid or its ester equivalent and a hydroxy terminated polyether polyol or other aliphatic or alicyclic diol. The copolymerization of these monomers may be carried out in a batch polymerization or preferably in continuous polymerization in the temperature range of 250°C to 300°C.

The strength of the copolyester drawn twisted yarn of the present invention compared with conventional polyester yarns is shown in Table I. The % shrinkage was in the range of 6% to 10%. The yarn was made into knitted hose and dyed. The dyed hose samples were measured for color strength, washing fastness and their dyeability with conventional product was compared. The results are shown in Table II.

TABLE I

Mechanical properties of draw twisted copolyester yarn vs. standard draw twisted polyester yarn of 120 denier, 72 filaments.

	-	Copolye	ster yarns	Polyester yarns	
Sr.No.	Draw ratio	Tenacity (gpd)	% Elongation	Tenacity (gpd)	% Elongation
1	1.655	4.2	39.2	3.28	36.2

TABLE II

The knitted filaments were dyed with Foron Blue SBGL 200% (supplied by Clariant India) at boiling water temperature (100° C) for about 45 min, without using any dye carrier.

	Dye Index	Washing Fastness *	
		Staining	Change in color
Copolyester yarn	221	4-5	4-5
Standard Polyester yarn	100	4	4-5

<sup>\*</sup> Washing fastness was determined by using ISO II method

The present invention involves the use of a novel continuous polymerization technique with optimization as to the location of addition, sequence of addition and the mode of addition of the comonomers so that the present invention can lead to the easy dyeability with an optimum balance of dyeability and physical properties.

The copolyester staple fibers of the present invention were produced as foresaid, and their mechanical properties are shown in Table III.

TABLE III

Mechanical properties of 1.2 denier copolyester staple fiber

Sr.No.	Denier	Tenacity (gpd)	% Elongation	T10	%DHS
1	1.15	6.4	26.9	3.1	5.8

The dyeing was carried out using a disperse dye i.e., Foron Blue SBGL 200% (supplied by Clariant India) at boiling water temperature (100° C) for about 45 min, without using a dye carrier. The dye index value was 527 for the dyed fiber of the present invention as compared with 100 for standard polyester fiber. The dyed yarn or fabric produced pursuant to the present invention has a dye index greater than 100, and generally at least about 120 to 500.

The copolymer fibers of the present invention may be blended with other fibers, including synthetic and natural fibers. The copolymer as fibers of the present invention may be blended with cotton, particularly for making fabric used in the manufacture of clothing.

In one specific embodiment, the method of preparing the modified copolyester filaments includes copolymerizing polyethylene glycol and long chain aliphatic dicarboxylic acid into polyethylene terephthalate in the melt phase to form a copolyester composition. As will be understood by those having ordinary skill in the art, such copolymerizing may be conventionally achieved by reacting

ethylene glycol and either terephthalic acid or (e.g.) dimethyl terephthalate in the presence of polyethylene glycol, the dicarboxylic acid, and the hydroxy terminated polyether polyol. Continuous polymerization is also within the purview of the present invention.

Finally, filaments are spun from the copolyester composition. In addition, and importantly the resulting copolyester filaments may be dyed using disperse dyes at (low) temperature of about 100° C (212° F) and at atmospheric pressure.

The drawn novel copolyester fibres can be spun into yarn as 100% or it can also be blended with other kind of natural and synthetic fibers to form blended Yarns. In this regard, the drawn copolyester fibre is especially (suitable) for blending with cotton fibers, rayon fibers, polypropylene fibers, acetate fibers, nylon fibers, spandex fibers, conventional polyester fibers. The fabric can then be manufactured using various combination of copolyester yarn either as 100% or in blends with other natural and synthetic fibers.

The PET-modified copolyester can be spun into partially oriented yarns (POY). As will be understood by those having ordinary skill in the art, POY is often comprised of from tens to hundreds of intermingled filaments (e.g., between 30 and 200) that are extruded from a spinneret at speeds typically between about 2000 and 4000 meters per minute. The POY is then typically drawn to form a drawn yarn, (e.g., by draw texturing, flat drawing, or warp drawing). Thereafter,

the drawn yarn is formed into fabric, which is typically finished as well. As will be known by those skilled in the art, texturing can be effected in numerous ways, such as air jet, gear crimping, and false-twist techniques. The modified PET yarns of the present invention may be readily formed into fabrics on conventional handlooms and power looms.

It is to be understood that the foregoing description relates to illustrative embodiments of the invention. Various modifications may be made within the scope of the invention, and accordingly the invention is not limited by the foregoing illustrative description, but is defined by the adjoined claims.

### We Claim

- A copolymer comprising a terephthalic acid or its ester equivalent, an ethylene glycol, and a flexible long chain aliphatic dicarboxylic acid or its ester equivalent.
- The copolymer of claim 1, further comprising a hydroxy terminated polyether polyol.
- 3. The copolymer of claim 1, further comprising an aliphatic or alicyclic diol.
- 4. A fiber comprising a copolymer, said copolymer comprising a terephthalic acid or its ester equivalent, an ethylene glycol, and a flexible long chain aliphatic dicarboxylic acid or its ester equivalent.
- 5. The fiber of claim 4, further comprising a hydroxy terminated polyol.
- 6. The fiber of claim 5, wherein the polyol comprises a polyether polyol.
- 7. The fiber of claim 4, further comprising an alipthatic or alicyclic diol.
- 8. The fiber of claim 4, wherein the fiber is dyed, and the dyed fiber has a dye index greater than 100.

- The fiber of claim 4 wherein the fiber is dyed, and the dyed fiber has a dye index of at least about 120 - 500.
- 10. The fiber of claim 9, wherein the dyed fiber comprises a disperse dye.
- 11. A method for making a modified polyethylene terephthalate copolymer product comprising copolymerizing a mixture comprising a terephthalic acid or its ester equivalent, an ethylene glycol, a flexible long chain aliphatic dicarboxylic acid or its ester equivalent and a hydroxy terminated polyol.
- 12. The method of claim 11, further comprising forming fibers from the polymer, preferably in a continuous polymerization operation, and further comprising forming yarn from the fibers.
- 13. The method of claimed 12, further comprising dyeing the yarn and wherein the dyeing comprises dyeing the yarn with a dispersed dye at about 100° C.
- 14. The dyed yarn produced from the method of claim 13.
- 15. The dyed yarn, of claim 14, wherein the dyed yarn has a dye index greater than 100.

16. The dyed yarn, of claim 12, wherein the dyed yarn has a dye index of at least about 120-500.

17. The dyed yarn, of claim 12, wherein the yarn has a controlled shrinkage of 6 to 10%.

18. The dyed yarn, of claim 12, wherein the dyed yarn has a washing fastness comprising a staining of 4 and a change in color of 4-5 as determined by ISO Method II.

19. A drawn and twisted copolymer modified PET disperse dyed yarn, which dyed yarn comprises a dye index greater than 100 and a controlled shrinkage of about 6 to 10%.

20. The yarn of claim 19, wherein the dye index is at least about 120-500.

21. A woven or knitted fabric comprising drawn and twisted copolymer modified PET yarns, said fabric comprising a dispersed dye, said dyed fabric comprising a dye index of at least about 120-500.

Dated 21st day of November 2003

For Reliance Industries Ltd

′S.P.Sapra President

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